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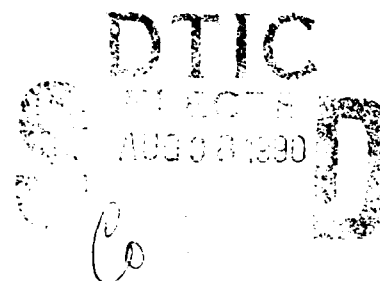


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# GROWTH AND CHARACTERIZATION OF III-V EPITAXIAL FILMS

Parke Mathematical Laboratories, Inc.

A. Tripathi and J.A. Adamski



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## FOREWORD

This report is the Annual Report for contract F19628-88-C-0061 covering the period March 4, 1988 through March 4, 1989.

The following individuals have been working on this contract for this reporting period:

Alok Tripathi : Parke Mathematical Laboratories, Inc.

Joseph A. Adamski : Parke Mathematical Laboratories, Inc.

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## SUMMARY OF OBJECTIVES

The general subject of this program is that of development of new or adapt existing methods for the preparation, growth and characterization of III-V electronic and optoelectronic materials for MOCVD technique. Investigations will be conducted on the growth of epitaxial layers using organometallic chemical vapor deposition method of selected III-V materials which are potentially useful for photonics and microwave devices. The following is a list of specific tasks to be accomplished during the contract period:

- a. Develop new or adapt existing III-V substrate preparation techniques to insure high mobilities and good morphology of the epitaxial layer grown on the substrate.
- b. Investigate the epitaxial growth characteristics of III-V single crystal epitaxial layers employing RADC metal organic chemical vapor deposition system.
- c. Develop new or adapt existing III-V epitaxial layer characterization method to determine the layer mobility, composition, thickness, and carrier concentration.
- d. Develop new or adapt existing characterization method to determine the quality of multilayered epitaxial structures for photonics and optoelectronic devices.

### **Work Done During This Period**

During this reporting period emphasis was placed on the testing for the safety, latelling, and understanding the operating procedures of the different components of the new RADC Metal Organic Chemical Vapor Deposition (MOCVD) system supplied by the CVD Equipment Corporation. Also, an attempt is being made to redesign and test part of the MOCVD machine.

This report contains the information on safety precaution and operating procedures for safe and efficient handling of the hazardous materials which is very important for the safe operation of the MOCVD crystal growth laboratory.

All the work was accomplished in close association with Dr. David W. Weyburne of RADC/ESM

## CHAPTER 1

### INTRODUCTION

#### Evolution of Gallium Arsenide Devices

Silicon has emerged as the most extensively used semiconductor material to fabricate devices due to the fundamental knowledge of its physical properties [1-12], to the almost perfect control of its preparation [13-20], and to the device fabrication technology [21-37].

Gallium arsenide and the related III-V compounds have been actively studied for two reasons. Some of their properties allow important improvements on the performances of classical devices; for example higher mobilities and larger gaps which allow for higher frequency and temperature operation [38,39]. Some characteristic features of III-V compounds give rise to new physical phenomena, such as high frequency electrical instabilities (Gunn effect) [40-51] and light emission by carrier recombination [52-54]. The high mobility and high saturated drift velocity (compared to silicon) have meant that GaAs semiconductor devices could operate at microwave frequencies [49, 51] where silicon devices are unable to function. The ability to produce semi-insulating substrates leads to lower parasitics and true monolithic circuit implementation [58, 59]. GaAs devices are more radiation tolerant than silicon MOS devices [60]. These phenomena have recently been used in such types of devices as microwave oscillators and amplifiers and light-emitting diodes and lasers [53-57]. The



future of these new compounds seems very promising especially for their application to optical telecommunications, in radar and for military devices. A commercial market also exists for direct satellite communication electronics and high speed computers. All these markets may be characterized by the need for large numbers of economical components operating above 1 GHz, and often above 10 GHz [61]. High speed logic is needed for electronic military applications and high-speed computers [62]. Many companies are actively exploring GaAs logic. GaAs devices can provide memory access times and logical operations at speeds several times greater than can silicon based devices. GaAs based devices such as the high electron mobility transistor (HEMT) promise performance equivalent to that expected from the far more complex superconducting Josephson junction technology [54]. GaAs has established a firm foothold in high-speed electronics and will certainly play a greatly expanded role in the near future. A recent review article lists over four hundred references on the intrinsic and major properties of GaAs [63] .

GaAs is grown in crystal ingots, then individual slices are sawed from the ingots by a diamond saw. These slices are lapped and chemi-mechanically polished to remove surface damage. For many GaAs device applications, these starting slices serve merely as mechanical support on which is first grown an upper conductive layer of appropriate thick-ness, resistivity and conductivity [64]. There are two primary methods of forming GaAs surface layers on GaAs substrates: epitaxy and ion implantation [64].

## **Epitaxy [64-78]**

Epitaxy consists of growing additional III-V materials on the surface of GaAs substrates in a manner that preserves the overall single crystal structure. Group III and V elements' atoms react with the crystal surface under temperatures, concentrations, and other conditions that result in surface crystalline growth. Dopants may be included among these atoms, and hence, incorporated into the crystal on the lattice sites.

Epitaxial layers are generally of higher crystal quality than the substrates they are grown on. If an active (doped) epitaxial layer is grown directly on the substrate, the impurities and defects in the substrate can degrade the crystalline properties and therefore the electrical properties, of the epitaxial layers. Therefore, buffer layers of undoped GaAs usually are epitaxially first grown on the substrate. The active layer is then grown on the buffer layer before the doping is introduced. In some cases, a top most layer of highly doped material may be grown to provide a contact layer.

There are three basic types of epitaxy that have been used for GaAs: liquid phase epitaxy (LPE), molecular beam epitaxy (MBE) and vapor phase epitaxy (VPE) [64].

### **Liquid phase epitaxy**

LPE is the oldest technique used to grow epitaxial layer on GaAs

crystals [65,66]. LPE involves the growth of an epitaxial layer on a crystalline substrate by direct reaction from the liquid phase. LPE remains a successful production technique for light emitting diodes and other structures that do not require thin ( $<1\mu$ ) and uniform epitaxial layers. Some of the major problems with LPE are the difficulty of growing uniform layers over large surface areas, the difficulty of avoiding growth instabilities and the necessity of wiping off the solvent after the layer is grown [74]. The technique is not suited for the growth of submicron layers which are required for high speed field effect transistors, microwave devices and integrated circuits. However, it remains an inexpensive method of epitaxy and is capable of growing many material compositions including GaAlAs [70, 71].

#### **Molecular beam epitaxy [77, 78]**

MBE is the major method developed for epitaxial growth. It can be described as a sophisticated evaporation technique performed in ultra high vacuum. In this procedure, the substrate is placed in a high vacuum and elemental species are evaporated from ovens, impinging upon the heated substrate. Here they assemble into crystalline order. With the proper control of sources (e.g. Ga, As, Al, Si, etc.), almost any material composition and doping can be achieved. Further, the layer thickness may be controlled with a resolution approaching one atomic layer. MBE's advantages are that it can produce thin layers with atomically abrupt junctions, and it can do so with high accuracy and uniformity across a substrate. The high vacuum requirements, complex

and costly equipment, slow growth rates and difficulty in growing films with elements such as P and N that have high vapor pressures are major disadvantages.

### Vapor phase epitaxy [64]

VPE has been the most popular form of GaAs epitaxial growth. In VPE, the Ga, As, and dopant atoms are brought to the substrate in the gas phase combined with other elements in molecules. Under appropriate conditions, reactions take place on the substrate surface that result in the desired atoms replicating the underlying crystal structure while the others form gaseous molecules that flow out of the system. The major VPE systems for growing GaAs have been chloride and/or hydride systems [79-86]. In the chloride system,  $\text{AsCl}_3$  is used as the arsenic source while in a hydride system  $\text{AsH}_3$  gas is used as the arsenic source (see ref. 64 for details on these systems). In both cases liquid gallium reacts with a chloride,  $\text{AsCl}_3$  or  $\text{HCl}$  to form  $\text{GaCl}$  which is transported to the growth zone. The advantage of these methods are that the epi-layer grown are thin and uniform compared to LPE. Also the throughput is high. The chloride and/or hydride systems can not be used for epitaxial growth containing Al compounds. It is also extremely difficult to grow layers with junctions.

Another category of VPE involves transport of group III elements by using volatile organometallics [87-90]. This procedure is designated OMVPE for OrganoMetallic Vapor Phase Epitaxy. It is also sometimes referred to as organometallic chemical vapor deposition

(OMCVD or MOCVD). This technique is becoming increasingly popular with industry because good quality epitaxial layers can be grown. The advantages of OMVPE are that it can produce a broad spectrum of epitaxial compositions with uniform layer thickness and abrupt junctions. It requires less costly equipment and can have a high growth rate. Another advantage of OMVPE is that it is a low temperature process as compared to hydride and chloride systems. The disadvantages include the need of excess of  $\text{AsH}_3$  or  $\text{PH}_3$  (as group V source) which are highly toxic, for example to grow InP the V/III ratio is as high as 500. Carbon contamination can also be a major problem, especially in compounds containing Al.

## CHAPTER 2

### GROWTH MECHANISM FOR III-V COMPOUNDS USING MOCVD

MOCVD is a vapor phase process using organometallics as starting materials. For the growth of III-V semiconductors, metal alkyls such as trimethylindium (TMI), Triethylindium (TEI) are typically used as the sources of the column III elements. When hydrides from the column V elements like arsine and phosphine are mixed with the metal alkyls at elevated temperatures in the vapor phase, reactions take place resulting in the deposition of single crystal layers of III-V compounds.

#### General Outline of Mechanism

The most general concept of the growth mechanics which is needed to be outlined is the sequence of events which take place in the reactor. Initially, the group III constituents are introduced into the gas stream by bubbling a ultrapure hydrogen carrier gas through a trimethylgallium (TMG) or triethylgallium (TEG) liquied sources. The vapor pressure of the source is exponentially dependant upon the temperature, hence the desired concentration of TMG or TEG can be controlled by varying the temperature and the hydrogen flow into the bubbler. The actual flow (cc/min.) of TMG into the reactor can be calculated using the formula:

$$F_{\text{TMG}} = \frac{\text{vapor pressure (TMG)}}{\text{pressure (reactor)}} \text{ (flow H}_2 \text{ into bubbler)}$$

The arsine concentration into the reactor can be directly manipulated by regulating the flow of AsH<sub>3</sub> from a 100% AsH<sub>3</sub> cylinder tank. Both the AsH<sub>3</sub> and TMG are carried to the reactor by the ultra-pure hydrogen. For a given system, the concentrations of the respective gases can be varied over a range of values; typically the mole fraction of the TMG is in the 10<sup>-4</sup> range, the mole fraction of the AsH<sub>3</sub> is in the 10<sup>-3</sup> range and the total flow of gas through the reactor might vary from 1 to 10 liters per minute. The next step requires the reactants to be transferred to the growth site. This takes place by diffusion and/or convection from the main gas stream to the wafer. After the reactants are absorbed at the surface the reaction takes place. At this point, the GaAs composes, it attaches to the existing substrate lattice, and the reaction by-products need to be desorbed from the surface and transferred back to the main gas stream. A summary of the steps taking place is given below:

1. Transport of reactants to epitaxial growth region
2. Transfer of reactants to crystal surface
3. Adsorption of reactants
4. Surface reactions
5. Desorption of products
6. Transfer of products to main gas stream
7. Transport of reactants out of epitaxial region

If these steps occur consecutively, then the slowest step will dominate the growth process; this is the rate limiting step. However if some of these steps occur simultaneously, then the fastest sequence will dominate the growth. It is possible that this parallel process occurs during the reaction/incorporation segments since it is not quite certain whether the constituents react and then move to a growth site or if they move to a growth site and then react.

Depending upon which steps of the sequence are dominating the process, the epitaxy can be under one of three different regions of control. The first, mass transport type I, occurs when the process is input rate limited. Here, it is assumed that the surface reactions and diffusion are in equilibrium and that the growth is governed by the rate at which the species move to and from the reaction site. The second, mass transport type II, is considered to be mass transfer limited. In this case, the diffusion of the reactant to the lattice regulates the growth process. Finally, if the sequence is most dependant upon the surface reactions, the system is under Kinetic control. When this occurs, the transport is occurring at a faster rate than the surface kinetics and therefore, the surface reactions are most important.

By varying the growth parameters, and monitoring the growth rate, one might be able to determine the region of control they are operating in. For instance, if the growth rate is strongly influenced by varying the reaction temperature, then the system is probably in the kinetic control regime. However, if the mole fractions of the gas

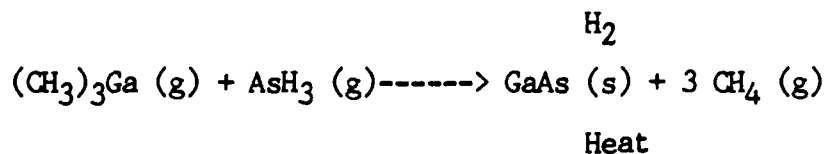


greatly effect the growth, then the system is mass transport limited. Looking at the effect of varying certain parameters is one technique for investigating the growth mechanics. And as is seen from the results, the growth process will probably shift from one type of control to another, depending upon the growth parameters.

The three major regions of the growth process are the input stage, where the reactants are introduced, the mixing stage, where possible adduct formation and elimination reactions take place, and the substrate surface, where growth proceeds.

### Reaction Thermodynamics

The MOCVD process is an inherently non-equilibrium situation. The concentration of reactants at the input is excessively high relative to the gas phase reactants at the surface which are in equilibrium with the substrate. This non-equilibrium state is the force which drives the reaction. Hence, the thermodynamics of the reaction should be considered first, since the ideal limit in the rate of crystal growth occurs then the entire system approaches equilibrium. The reaction of  $\text{AsH}_3$  and TMG to form GaAs may be written as:



Using the Laws of Thermodynamics, some insight can be attained to the

spontaneity of this reaction. If the change in Gibbs free energy ( G ) of a reaction is negative, then the reaction will occur spontaneously. The definition of G follows:

$$G = H - T S$$

where, H is the change in heat content, or enthalpy, and S is the change in entropy of the system. The entropy is a measure of randomness, and in order for a reaction to be spontaneous, there must be a net increase in entropy. If H is defined as:

$$H = H_f (\text{products}) - S (\text{reactants})$$

Then, one can calculate the change in Gibbs free energy for the reaction of TMG and AsH<sub>3</sub>.

For:

$$G = G_f(\text{GaAs}) + 3 G_{P_f} (\text{AsH}_3) - G_f (\text{TMG})$$

$$G (298 \text{ K}) = -80.48 \text{ KCal/mole}$$

$$G (700 \text{ K}) = -111.6 \text{ KCal/mole}$$

$$G (1000 \text{ K}) = -114.4 \text{ KCal/mole}$$

$$G (1300 \text{ K}) = -86.3 \text{ KCal/mole}$$

According to the thermodynamics, the reaction of AsH<sub>3</sub> and TMG is spontaneous over a large temperature range.

Another parameter to be considered is the thermodynamic equilibrium constant (K). This constant expresses what the ratio of the products partial pressure is to the reactants partial pressure at equilibrium. The magnitude of K gives us insight as to the extent to which the reaction proceeds toward completion. The equilibrium constant of the experiment is given by:

$$K = \frac{(P_{CH_4})^3}{(P_{AsH_3})(P_{TMG})}$$

Since the density of solid GaAs does not change throughout the reaction, the GaAs(s) is not included in the numerator of the expression; rather, it is constant which is included in K. The change in the Gibbs free energy can also be written as a function of K where:

$$G = -2.303 RT \log K$$

There are three major parameters which strongly effect a reaction at equilibrium; they are reactant concentration (Reactant Partial pressure), Reaction Temperature, and Catalysts. By changing the reactants partial pressure, the system is forced away from equilibrium, causing the reaction to be driven back toward the equilibrium position. When the temperature is varied, the equilibrium constant changes, again causing the system to be in nonequilibrium. And, when a catalyst is introduced, the energy necessary for the reaction to proceed is lowered. Although the GaAs growth process

exists far from equilibrium conditions and strays severely from the ideal thermodynamic principles, we will see that these three parameters which greatly effect the reaction also strongly effect the GaAs growth process.

### Homogeneous Gas Phase Reactions

The state of the reactants and products in the gas phase has been a topic of vast uncertainty. There are three major phenomena which need to be investigated, adduct formation, polymer formation, and decomposition vs. composition.

An adduct forms when an electron acceptor molecule (Lewis Acid) combines with an electron donor molecule (Lewis Base). In the case at hand, it seems possible that a certain amount of the  $(CH_3)_3Ga$  and  $AsH_3$  form adducts. The amount of adduct formation depends primarily on how extensively the two molecules are mixed at low temperatures. However, an important aspect of the adducts in the gas phase is not how it forms, but how it decomposes. Since the bond energy is low, the adduct could just break apart into TMG and  $AsH_3$  as it moves further into the high temperature region in the immediate vicinity of the susceptor. This process would have very little effect on the reaction mechanisms. More importantly though, the adduct could go through an elimination reaction as it approaches the wafer. If this occurs, the  $R_3III:R_3'V$  molecule sequentially loses  $RR'$  groups until a very strongly bonded, highly reactive  $RIII:RV$  polymeric molecule remains (where the R represents the methyl group and the  $R'$  represents the

hydrogen for the GaAs reaction). Since there are two dangling bond sights, the RIII:RV readily forms (RIII:RV)<sub>n</sub> polymers if possible. Although adduct and polymer formations in GaAs OMCVD might be limited, it should be considered since the RIII:R'V molecule could be a source of carbon incorporation into the lattice.

The other aspect of the gas phase reactions involves the decomposition or pyrolysis of the various III-V constituents. Many researchers feel that the TMG molecule and the AsH<sub>3</sub> molecule become pyrolysed as they enter and pass through the boundary layer. D.H. Reep [1] has calculated that the activation disassociation energies for the three GaCH<sub>3</sub> bonds are 59.5, 53.4, and 77.5 Kcal/mole, respectively. Hence, it is thought if there is enough energy, the first two methyl groups will be released and a tightly bonded GaCH<sub>3</sub> complex will remain. Much of the general acceptance of this theory lies in the experimental data which presumably supports this concept.

I.R. spectroscopy by various groups has shown there to be no TMG in the boundary layer region. In particular, M.R. Leys and H. Veenliet used in-situ I.R. spectroscopy where the sampling probe was 0.2 cm above the substrate. It is concluded that at a temperature above approximately 600° C, all the TMG in the boundary layer has been decomposed. Also, Leys and Veenliet point out that the sensitivity of the spectrum for TMG is about 1% of the TMG at the input ( $P_{\text{TMG}}$  at input =  $1 \times 10^{-4}$  atm). J. Kishizawa and T. Kurabyashi have also done extensive I.R. spectroscopy on AsH<sub>3</sub> and TMG + AsH<sub>3</sub>. It is shown that the TMG approaches 100% decomposition at about 600°C while the AsH<sub>3</sub>

does not decompose nearly as much.

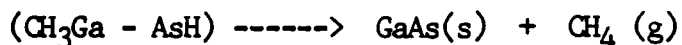
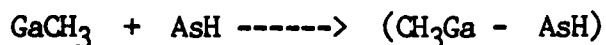
However, when the TMG is introduced to the  $\text{AsH}_3$ , the  $\text{AsH}_3$  decomposition is enhanced, and when a GaAs substrate is included, the decomposition of  $\text{AsH}_3$  progresses even further. The availability of the methyl groups seem to be responsible for the increased  $\text{AsH}_3$  decomposition, while the GaAs substrate seems to act as a catalyst. Much of this data implies that the pyrolysis of the  $\text{AsH}_3$  and the TMG is a major step in the growth mechanics.

Finally, it has been found that under the proper growth conditions an appreciable amount of TMG and  $\text{AsH}_3$  is still available in the vapor phase at the substrate. Considering the high linear velocities of the gases and the time necessary for 100% decomposition of TMG and  $\text{AsH}_3$ , the concentration of TMG and  $\text{AsH}_3$  in the gas phase must be considered.

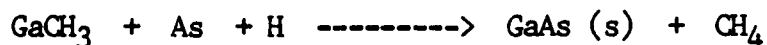
Needless to say, the homogeneous gas phase reactions in MOCVD are difficult to precisely predict; given the strong dependence on temperature and the temperature gradient across the reaction chamber, it is difficult to identify which species are dominating the gas phase composition. Some possibilities are,  $(\text{RIII.R'V})_n$  polymeric compounds,  $(\text{GaCH}_3)_n$  polymeric compounds,  $\text{AsH}_2$ ,  $\text{AsH}$ , TMG and  $\text{AsH}_3$ . By implementing several sequences of crucial experiments (possibly including some form of spectroscopy), the gas phase composition could be better defined than it is to date.

## Surface Reaction

Given the information on the gas phase composition, there are three general theories for the surface reaction. The first two assume that the gas phase is dominated by  $\text{GaCH}_3$  molecules and various forms of As constituents as explained by the pyrolysis concept. Here the  $\text{GaCH}_3$  and AsH molecules are absorbed at the surface, then the reactions between the two molecules transpire and the  $\text{CH}_4$  by-product is desorbed from the surface. This process can be represented as follows:



The second alternative being that the  $\text{GaCH}_3$  is absorbed at the surface, and then diffuses to the appropriate Gallium kink site, where it forms GaAs. This may be represented by :



Hence, this process is enhanced by the concentration of arsenic. The final possibility postulates that an abundance of TMG and  $\text{AsH}_3$  exists at the substrate surface. Now as the TMG collides with the  $\text{AsH}_3$  at the surface, the complete reaction occurs, forming GaAs and Methane.



All of these surface reactions are assuming ideal thermodynamic conditions. In reality, under certain conditions, the thermodynamics may not be as desirable where upon incomplete reactions may occur.

### Boundary Layer Diffusion

In the 600-800°C range, it is generally considered that the reaction mechanisms are not the rate limiting step of the growth process. In this range, the diffusion of the species through the boundary layer to the surface proceeds slower than the surface reaction and is therefore the limiting step of the growth process. Hence, the growth rate is almost entirely controlled by the diffusion mechanisms.

The boundary layer is the sliver of the gas stream above the wafer that the constituents diffuse through in a very short period of time. It is not actually a stagnant region above the substrate, however, such a concept helps to visualize the diffusion process. Obviously, if the boundary thickness varies over the growth area, then the diffusion time will vary and the growth rate will not be uniform across the substrate.

Since the ratio of the group V partial pressure to the group III partial pressure is usually large (at least 10), then the diffusion of the group III molecules limits the growth rate. The flux of the group III molecules may then be written as:



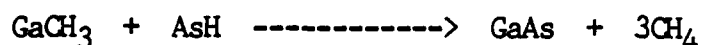
$$J_{III} = D_{III} (P^0_{III} - P_{III})/RTd$$

where  $D_{III}$  is the diffusion coefficient in the gas stream and it is dependent upon  $T^2$ ,  $P_{III}$  is the partial pressure at the growth interface and  $d$  is the boundary layer thickness. As the boundary layer thickness is decreased or the reactant partial pressure is increased, the flux will increase, therefore increasing the growth rate.

## EFFECTS OF TEMPERATURE, PRESSURE, TOTAL FLOW, MOLE FRACTIONS, AND SUBSTRATE:

In this section the general effect on the growth process of varying the major growth parameters in the MOVPE system is considered.

Varying the temperature of the susceptor can strongly effect both the surface reactions and the gas phase reactions. In the gas phase the temperature greatly influences the amount of TMG and AsH<sub>3</sub> decomposition. Also, the temperature and the magnitude of the temperature gradient can greatly effect the formation of adducts and polymers. Obviously, the extent of molecular decomposition and polymer formation would severely effect both the



reaction mechanisms. At low temperatures, the effect of reduced reaction rates, incomplete reactions, and less decomposition might be so great that the system would shift from diffusion limited growth to kinetically controlled growth, while at the high temperature end, increased deabsorption from the surface, and extensive decomposition, again might precipitate kinetically controlled conditions.

The direct effects of total pressure probably has very little effect on the reaction mechanisms since the partial pressures are the

dominating aspect in the reaction. However, by reducing the total pressure, the linear gas flow velocity is increased. This change in linear velocity definitely will influence the boundary layer thickness and diffusion rate since the flux rate is proportional to the square root of the velocity. Also, increasing the velocity will reduce the amount of time the molecules spend in the high temperature zone. Obviously this should effect both the extent of molecular decomposition and the completion of various gas phase and surface reaction.

Changing the total flow, again, will change the linear velocity of the gases. Hence, changing the total flow should give some similar results to changing the total pressure.

Varying the mole fractions (or partial pressures) of the input gases probably has the strongest influence on the reaction mechanisms. The concentration of the constituents in the gas phase strongly affects the availability of all the different forms of the group III and V reactants at the substrate. These changes will drastically affect both impurity incorporation and growth rates. Also, the partial pressures of the reactants will determine the boundary layer diffusion rates; hence, the mole fractions will strongly affect those systems which are in the mass transport I and II regime.

Finally, the substrate can be considered as a heterogeneous catalyst. Primarily, the substrate acts as a site for the reaction to occur. The large energy liberation of the reaction needs to be

transferred to something; the substrate is an ideal sink for the generated reaction energy. Also, if the molecules lightly bond with the dangling bonds on the substrate surface, the reactants molecular bonds and bond energies will be stretched, and distorted. This would catalyze the reaction, affecting both its rate and completeness.

It must be kept in mind that parameters which effect the reaction mechanisms may be masked out when the system is in the mass transport regime. This is not to say they are not important, since the reaction mechanisms can influence both the growth rate and impurity incorporation.

#### **IMPURITY INCORPORATION:**

One of the driving forces in studying MOVPE growth mechanics is the incorporation of impurities. There are many intrinsic impurities which are generally found in MOVPE GaAs; a few of the primary impurities are, silicon, germanium, zinc, and carbon. From the data it appears that the donor incorporation can be controlled by adjusting the temperature and the V/III ratio. On the other hand, the carbon in the films generally increases at low ratios and at elevated temperatures. It is conceivable that the carbon is directly incorporated into the lattice either when incomplete surface reactions occur or when carbon containing polymeric complexes react at the surface. Depending upon which molecules dominate the gas phase reactions as well as which surface reaction is most prevalent, the incorporation of the carbon can be explained. Hence, more

experimental research needs to be done to determine the dominating phenomena in the gas phase and at the surface.

## CHAPTER 3

### APPARATUS

The RADC/ESM'S MOCVD system consists of four major components:

- a. The gas handling system which includes the group III source alkyls, group V source hydrides, dopants, all of the valves, plumbing, and instruments necessary to control the gas flows and mixtures.
- b. The reaction chamber in which the pyrolysis reaction and deposition occur.
- c. The heating system used to obtain pyrolysis temperatures.
- d. The exhaust and the low pressure pumping systems.

Each component of the MOCVD system is discussed below:

#### a. Gas handling system

The purpose of the gas handling system is to deliver precisely metered amounts of uncontaminated reactants without transients due to pressure or flow changes.

Pd-purified hydrogen gas is used as the carrier gas. The inert nitrogen gas is used for purging purposes and also for actuating the air operated valves.

The group III alkyls are held in temperature controlled baths and electronic mass flow controllers are used to regulate the carrier gas. The group V hydrides are introduced as dilute mixtures (5-10%) in hydrogen, but there is a provision for using 100% concentrations of these hydrides. Again electronic mass flow controllers are used to meter the reactants. The dopants can be used either as alkyls or hydrides.

Each of the reactants is fed into a common manifold, with alkyls, hydrides and dopants separated until introduction into the reactor. The valving provisions are employed to switch the sources from reactor manifold to a bypass manifold. This allows the intervening piping to fully purge before injection into the reactor, and it also allows injection into the reactor to be terminated without flushing excessive lengths of tubings. All the gas handling system is assembled from stainless steel tubing and valves. All the valves are automated and interfaced with computer, as the mass flow controllers. Manual operation is possible, but the requirements of complex multilayered and graded structures make non-automated systems difficult to use reliably.

The gas handling manifold for the low pressure system has a control valve between the manifold and the low pressure reaction

chamber which allows the gas handling system to operate at fixed pressure independent of reaction chamber. This is done because it is essential to maintain a constant pressure over the group III alkyl source since this pressure influences the vapor concentration in the gas phase.

#### **b. Reaction chamber**

The RADC MOCVD system has a vertical reaction chamber. In this vertical reaction chamber, the reactants are introduced from the top. The substrate wafer lies flat on top of a moly susceptor which can be rotated at different speeds. The reactor has a 1/4" inject tube made of quartz which is used to spread the flow of the inlet reactants. To decrease the possibility of gas contaminating spent reaction products to recirculate in convection currents and to become entrained in the inlet stream, the inject tube is placed 1" above the moly susceptor. The reaction chamber is cooled by a chilled water jacket placed around it.

#### **Leak checking of the gas handling system**

The source reactants used in the MOCVD system are highly toxic, ultra pure and reactive gases. It is therefore necessary to leak check the system when new, and again after any equipment replacement in the gas distribution system. A helium leak detector capable of detecting leaks in the range of  $1 \times 10^{-6}$  cc/sec of helium is used for leak checking the system.



Using the schematic diagram showing gas flows and relative component placement all the gas distribution system is labelled and the flow directions are marked. This marking is used as reference during leak test.

**Never use a leak detector on a gas transfer line filled with a reactive gas without purging completely with several purge cycles using Nitrogen as purge gas.**

Preliminary leak test of sections of the system is done by checking for pressure decay over 5 minute time periods. Then each section is -tested for leak using helium leak detector. The procedure is given below:

#### **A. Leak check of PH<sub>3</sub>-A Line**

Note : Close the PH<sub>3</sub> tank.

##### **a. Source line:**

###### CLOSE Valve

Hand valve (HV) 21

HV 6

HV 7

###### OPEN Valve

HV 20

HV 4

HV 5

Pump down using helium leak detector and perform leak test. This procedure tests for leak in the plumbing from PH<sub>3</sub> tank to the MOCVD

system.

**b. Vent line:**

CLOSE Valve

HV 21

HV 4

HV 6

Air operated valve (AOV) 8

OPEN Valve

HV 20

HV 5

HV 7

AOV 9 (SW9)

Note : AOV 8 ==> SW 8 & AOV 9 ==> SW 9

SET

MFC 9 TO 200 sccm

AOV 38 TO "VENT"

SWITCH (SW) 54 TO "OFF" POSITION

Pump down using leak detector and perform the leak test.

**c. Inject line:**

CLOSE Valve

HV 21

HV 4

HV 6

AOV 8 (SW 8)

OPEN Valve

HV 20

HV 5

HV 7

AOV 9 (SW 9)

SET

MFC 9 TO 200sccm  
AOV 38 TO INJECT  
SW 54 TO "OFF" POSITION  
SW 51 TO "OFF" POSITION

Perform the leak test using the helium leak detector.

**B. Leak check of PH<sub>3</sub>-B line**

Note : Close the PH<sub>3</sub> tank.

**a. Source line:**

CLOSE Valve

HV 21

HV 10

HV 11

OPEN Valve

HV 20

HV 8

HV 9

Perform the leak test.

**b. Vent line:**

CLOSE Valve

HV 21

HV 8

HV 10

AOV 14 (SW 14)

OPEN Valve

HV 20

HV 9

HV 11

AOV 15 (SW 15)

SET

MFC 12      TO      200 sccm  
AOV 41      TO      "VENT"  
SW 54      TO      "OFF" POSITION

Test for leak using helium leak detector.

**c. Inject line:**

CLOSE Valve

HV 21  
HV 8  
HV 10  
AOV 14 (SW 14)

OPEN Valve

HV 20  
HV 9  
HV 11  
AOV 15 (SW 15)

SET

MFC 12      TO      200sccm  
AOV 41      TO      "INJECT"  
SW 54      TO      "OFF"  
SW 51      TO      "OFF"

Test for leak.

**C. Leak check of AsH<sub>3</sub> line**

Note : Close the AsH<sub>3</sub> tank.

**a. Source line:**

CLOSE Valve

HV 21

HV 14

HV 15

OPEN Valve

HV 20

HV 12

HV 13

check for the leak.

**b. Vent line:**

CLOSE Valve

HV 21

HV 12

HV 14

AOV 12 (SW 12)

OPEN Valve

HV 20

HV 13

HV 15

AOV 13 (SW 13)

SET

MFC 11 TO 200 sccm

AOV 40 TO "VENT"

SW 54 TO "OFF"

**c. Inject line:**

CLOSE Valve

HV 21

OPEN Valve

HV 20

HV 12

HV 14

AOV 12 (SW 12)

HV 13

HV 15

AOV 13 (SW 13)

SET

MFC 11 TO 200sccm

AOV 40 TO "INJECT"

SW 54 TO "OFF"

SW 51 TO "OFF"

**D. Leak check of HCl line**

Note : Close the HCl gas tank.

**a. Source line:**

CLOSE Valve

HV 21

HV 18

HV 19

OPEN Valve

HV 20

HV 16

HV 17

Pump down and leak check.

**b. Inject line:**

CLOSE Valve

HV 21

HV 16

OPEN Valve

HV 20

HV 17

HV 18

AOV 18 (SW 18)

HV 10

AOV 19 (SW 19)

SET

MFC 14      TO    100sccm

SW 49      TO    "OFF"

Note : There is no vent line for HCl.

## CHAPTER 4

### HYDROGEN PURIFIER

#### OPERATING PRINCIPLES

The Palladium Diffusion Process purifies gaseous hydrogen in a purity beyond the limits of any known means of detection. The purification is accomplished by taking advantage of a property of a palladium-silver alloy which exhibits selective permeability to hydrogen gas. The hydrogen gas will diffuse through the palladium-silver alloy but other gases will not. The metal is impervious to any impurity.

The rate of hydrogen diffusion:

1. Varies directly with the surface area of the palladium alloy.
2. Varies exponentially with the temperature, increasing at a decreasing rate as the temperature increases.
3. Varies directly with the difference between the square roots of the partial pressures of hydrogen on both sides of the palladium alloy tubes.
4. Varies inversely with the thickness of the palladium alloy septum in the direction of flow.



In service, under operating conditions, the pure hydrogen flow rate is adjusted and controlled by the inlet pressure. The hydrogen purifier contains a fixed length of tubing of definite wall thickness so that the surface area and septum thickness are constant. By controlling the operating temperature to  $400^{\circ}\text{C}$ , the temperature variable is eliminated. (This temperature of  $400^{\circ}\text{C}$  is the optimum operating temperature which will give the highest output of pure hydrogen consistent with long life of the palladium alloy tubes). The difference between the square roots of the partial pressures of hydrogen on both sides of the tubes is the only remaining variable affecting the pure hydrogen flow rate. The flow rate of pure hydrogen then is adjusted with the pressure of the impure hydrogen entering the purifier. The pressure of pure hydrogen can also adjust the flow rate, however, under operating conditions it is usually held constant. The inlet pressure is limited by the physical strength of the tube wall and should not exceed 200 psig.

Poisoning of the palladium-silver alloy by impurities is of no concern if a commercial cylinder hydrogen source is used. However, when other sources of impure hydrogen are used, which contain large amounts of carbon tetrachloride, hydrogen sulfide, or other gases containing sulphur, poisoning takes place at a rapid rate. In these cases, it is necessary to remove the poisoning gases by external means before admitting the gas to the purifier. Poisoned palladium-silver tubes are easily regenerated with air as the inlet gas at operating temperature to oxidize the impurities and then with hydrogen to reduce the metal oxide formed on the tubing wall.

In order to prevent impurities from accumulating on the impure side of the palladium alloy septum and thereby decreasing the partial pressures of hydrogen, they are bled out in a controlled stream of impure hydrogen and either burned or innocuously vented.

The purifier temperature is regulated by an electrical heater which is controlled by the operating thermoswitch.

The cabinet contains a blower which serves the dual purpose of pulling a stream of cool air through the outlet gas cooling coil and of preventing an accumulation of hydrogen within the cabinet in the event of a leak.

## OPERATING INSTRUCTIONS

1. Close valve 2 (SW-2) and valve 5 (SW-5). Open valve 1 (SW-1), valve 3 (SW-3) and valve 4 (SW-4) to purge impure  $H_2$  lines with nitrogen. Make sure that the pure  $H_2$  line has a vacuum of 30".
2. Raise the temperature of the furnace to  $385^{\circ}C$  with  $N_2$  flowing through it. This helps in getting rid both the impure and pure side of any gases which might form an explosive mixture with hydrogen during start up. The maximum operating temperature should never exceed  $400^{\circ}C$ .
3. Once the temperature stabilizes at  $385^{\circ}C$ , close valve 1 (SW-1), valve 3 (SW-3) and valve 4 (SW-4). Make sure that the vacuum in pure hydrogen line is 30" and it is holding.
4. Open "inlet" valve 2 (SW-2) to admit the impure hydrogen.

Note : The flow of pure hydrogen may be adjusted by the pressure of the inlet gas since the diffusion rate of pure hydrogen is dependent on the difference between the partial pressures of the hydrogen on both sides of palladium tubes. Therefore the pure hydrogen flow rate can be varied by adjusting the differential between inlet and pure outlet pressure. The recommended inlet pressure is 200 psig or less. The unit should never be subjected to a pressure higher than 250 psig.

5. Open needle valve 1 (NV-1) and adjust it to pass approximately 5% of the "inlet"  $H_2$  gas flow rate.
6. Discard the hydrogen purified during the first few minutes of operation which may be slightly contaminated.

### **TEMPORARY SHUT-DOWN**

If shut down is only for a short time it is advisable not to allow the H<sub>2</sub> purifier to cool down.

1. Close "inlet" valve 2 (SW-2) and needle valve 1 (NV-1) and close valve 5 (SW-5) so that approximately 10 psig of gas pressure is trapped in the purifier.
2. Leave power on so that the furnace temperature is maintained at 385°C.

### **START-UP FROM TEMPORARY SHUT-DOWN**

1. Open "inlet" valve 2 (SW-2), adjust the needle valve 1 (NV-1) and open valve 5 (SW-5).

### **COMPLETE SHUT-DOWN**

1. Close the "inlet" valve 2 (SW-2) and after the gas had dropped to 10 psig close the needle valve 1 (NV-1).
2. Close the pure hydrogen gas valve 5 (SW-5).
3. Purge the purifier by opening the valve 3 (SW-3) and valve 4 (SW-4) so that the pure hydrogen line reads 30" of vacuum.

4. After 10 minutes open valve 1 (SW-1) with venture pump.
5. Switch off the H<sub>2</sub> purifier heater.

## SPECIAL PRECAUTIONS

Hydrogen is a highly explosive material when mixed with air. Extreme care should be taken when connecting external fittings to make certain they are leak tight. Periodic checking of the fittings for leak is advisable. The hydrogen diffusion cell should give years of good service provided the following safe operating precautions are taken :

- Never subject the purifier to a pressure higher than 250 psig. The recommended operating pressure is 200 psig or less.
- Never allow the pressure on the pure side of the system to exceed the pressure on the impure side of the system as this may cause the tube to collapse.
- Filter the inlet gas if it contains solid or liquid impurities.
- Never start the hydrogen flow unless all air has been purged out of all impure  $H_2$  lines with nitrogen and the unit has been completely evacuated so as to avoid the possibility of explosive mixtures forming.
- It is very important that the valve 1 (SW-1), valve 3 (SW-3), and valve 4 (SW-4) be closed while hydrogen is being purified.
- The maximum recommended operating temperature is  $400^{\circ}C$  ( $750^{\circ}F$ ).

Never operate the purifier at a temperature higher than 400°C.

. The diffusion cell should be operated in well-ventilated area.

. Do not use any instrument containing mercury for any purpose in connection with the hydrogen purifier. Mercury vapor can completely and permanently poison the palladium-silver membrane.

1. In order to avoid corrosive damage to internal parts, insure that corrosive contaminants such as halogens, halides, sulphur compounds, and metal compounds or vapors are absent from the hydrogen feed gas.
2. Oxygen in a concentration greater than 1000 ppm must never be allowed to enter a diffusion cell containing hydrogen. Oxygen can be eliminated by installing a Oxygen Removing Purifier in the feed line upstream from the diffusion cell.
3. Always heat up and cool down your diffusion cell in the absence of hydrogen. This can be done by evacuating or by purging with inert gas.
4. Sulfur compounds, unsaturated hydrocarbons, free carbon, iron, lead, mercury, zinc, halides, oil and grease will poison the precious metal membrane in your diffusion cell and should be entirely eliminated from the feed gas.



## TROUBLE SHOOTING

### A. NO POWER:

1. Check fuse.

### B. OUTPUT OF HYDROGEN DECREASING:

1. Check for proper adjustment of bleed gas valve (Needle valve - 1). Too low a bleed gas flow rate will cause accumulation of impurities on the impure side and thereby decrease the pure hydrogen output.
2. Check for a decrease in the hydrogen content of impure gas.
3. Check for poisoning impurities in the feed gas.
4. Regenerate the palladium-silver alloy by passing air through the impure side. Then pass hydrogen through the impure side and original pure hydrogen output should be regained.

Note: It is advisable to keep periodic record of the pure hydrogen output flow rates obtained under the normal operating conditions so that any sign of poisoning can be easily detected by comparison.

## REGENERATION PROCEDURE OF PALLADIUM DIFFUSION CELL

During normal operation, it is possible for carbonaceous matter to accumulate on the diffusion cell wall. This may be due to traces of residual oil in commercial cylinders, and various other sources. As palladium is normally a highly active oxidation catalyst, a simple air regeneration will normally bring the diffusion cell back to its initial state. Regeneration would be accomplished in the following steps:

1. Completely evacuate the unit as in normal start-up.
2. Heat the unit up to its normal operating temperature (400°C).
3. Shut off vacuum and run commercial nitrogen through the unit at relatively low pressures, i.e., 10psig.
4. Gradually add air to the nitrogen purge stream. For example, put 20cu. ft./hr. of nitrogen through the diffusion cell and add 1-2 SCFH of air. The exact amount is not a critical matter. After running for 15-30 minutes, gradually increase the air and decrease the nitrogen until air is passing through the diffusion tubes.
5. Shut off air, evacuate the unit, purge with nitrogen, and return to the normal operating procedure.

## CHAPTER 5

### OXYGEN REMOVING PURIFIERS INSTALLATION AND OPERATION INSTRUCTIONS

Oxygen Removing Purifiers (ORP) catalytically combine hydrogen and oxygen to form water vapor. The water vapor passes off with the purified gas and is removed by Hydrogen Purifier.

THEORY - ORP contain a precious metal catalyst that promotes the reaction of hydrogen and oxygen gases to form water vapor, normally at ambient temperature. The two gases combine in a stoichiometric ratio of two units of hydrogen to one unit of oxygen. In order to completely eliminate hydrogen or oxygen, the other gas must be present in excess (roughly 110%) of the stoichiometric ratio to ensure the reaction is driven to completion.

CONSTRUCTION - ORP is made of seamless stainless steel tubing. The tubing is packed with high-surface-area ceramic pellets coated with a very active precious metal catalyst. The end caps are welded on using an inertia weld, which forms a grain-refined solid-state bond with no grain growth, cast structure, porosity, segregation, or filler materials.

INSTALLATION - ORP must be installed vertically to prevent channeling of the gas after the catalyst has settled. The inlet gas should always enter through the top of the unit. Up-flow may fluidize the catalyst bed and cause attrition of the catalyst. Metallic pipe or tubing

should be used to install the OR. Plastic or rubber tubing may allow the purified gas to become recontaminated. An inert material such as Teflon tape should be used for sealing threaded connections.

OPERATION - Both connections to the ORP should be purged and pressure tested for leaks with an inert gas such as argon or nitrogen before placing the unit in service. Once the system has been purged and pressure tested, the gas flow can be turned on and the OR will begin to work immediately.

The ORP generally operate at room temperature; however, it may be necessary to heat the gas stream if it is near its dewpoint, or if carbon monoxide is in the feed gas. The ORP should not be operated above 1300°F. The ORP is designed to be operated continuously at elevated pressure (see the ORP label for maximum pressure rating). Pressure drop across the ORP is negligible.

The reaction of hydrogen and oxygen is an exothermic reaction (heat releasing). The temperature rise associated with the reaction is approximately 30 F per 0.1% of oxygen removed from hydrogen or nitrogen and 40 F per 0.1% of oxygen removed from argon or helium. The actual temperature rise depends upon the heat capacity of the gas being processed and on heat losses from the system.

## CHAPTER 6

### MPS-190 SPECIALTY GAS HANDLING SYSTEMS

#### INTRODUCTION:

The handling of the process gases used in the semiconductor industry presents a technical challenge because of the special characteristics of those gases: toxic, reactive, corrosive, high purity. This has emphasized the importance of improving the handling methods with a view to providing safety to personnel, protection of the physical process, efficiency of the operation by preserving the high purity of the process gas.

Typical phases involved in the handling of the process gases include:

- . Attaching a gas cylinder to the manifold of a delivery system.
- . Putting the cylinder in service to supply the manifold.
- . Isolating the process from the delivery system.
- . Disconnecting a gas cylinder from the manifold.

A common purpose through those phases is to achieve the following goals:

- . Prior to disconnecting a cylinder from the manifold, exhaust in a safe manner the process gas contained in the manifold and purge the manifold with an inert gas until the concentration of the process gas is reduced to the required safety limit.
- . Prior to putting a new cylinder in service (opening the cylinder valve to supply the manifold), purge the manifold with an inert gas to insure that the concentration of the remaining impurities is not detrimental to the level of purity required for the process gas.

This establishes the need for an effective purging method which will remove the process gas from the manifold and substitute for it a high grade inert purge gas free of impurities.

Two basic approaches are used in practice. One consists in flushing by applying the purge gas at one end of the manifold and creating a continuous flow which is exhausted at the other end of the manifold. This removes the process gas by displacement and entrainment. The other approach uses the principle of dilution: the manifold is successively vented to a low pressure (atmospheric level or below atmospheric level), pressurized with purge gas to a pressure equivalent to several atmospheres, then vented again to the low pressure level. The cycle is repeated several times. At the end of each cycle the concentration of the process gas is diluted by a factor which is directly related to the ratio of the vent pressure to the

purge gas pressure. The principle suggests that the combination of number of cycles and dilution factor will allow to reduce the concentration of the process gas to a very small value. It suggests also that it is independent of the configuration of the manifold and of the path taken by the purge gas flow.

#### **DESCRIPTION:**

##### **1. Purge by Dilution:**

A manifold which is filled with a process gas is vented to atmospheric or subatmospheric level. A purge gas is applied to the manifold until the pressure in the manifold reaches a nominal preset pressure, then the manifold is again vented to atmospheric or subatmospheric level. The successive phases of pressurizing and venting constitute a purging cycle. In the course of the cycle the concentration of the process gas has been reduced by "injecting" into the manifold a greater volume of purge gas than the initial volume of process gas. If the addition of purge gas brings the pressure from atmospheric level to 60 psi, the manifold will contain one volume of process gas and four volumes of purge gas: the concentration of the process gas will be reduced to  $1/5$  of its initial value. The dilution factor is  $1/5$ : it is equal to the ratio of the vent absolute pressure over the absolute pressure created by the purge gas.

The purging cycle is repeated several times. At the conclusion of each cycle the process gas concentration is reduced to  $1/5$  of its

previous value. At the end of N cycles the concentration will be reduced to  $(1/5)^N$  of its starting value.

It is apparent that the total dilution is directly related to the number of cycles N and to the dilution factor obtained at each cycle. The dilution factor can be increased by pressurizing at a higher pressure and venting at a lower pressure.

## **2. Purge by Flush-Flow:**

One end of the manifold is connected to a purge gas supply, the other end is connected to the vent line. The purging operation consists in applying a continuous flow through the manifold for a given length of time to displace the process gas and replace it with purge gas. The removal of the process gas proceeds through a combination of turbulent mixing and entrainment. It is evident that direct contact between the two gases is required for an effective operation. It suggests that the effectiveness will be seriously affected by any dead ended side capacity.

## **3. MPS-190 Purge Panel Operating Instructions:**

The following procedures have been devised for the safe operation and manipulation of the hazardous gas purge panels which are installed in the laboratory for handling arsine and phosphine.

In general, changing of hazardous gas cylinder (or metalorganic bubblers) will be done by the "buddy" system and with appropriate



safety apparatus and precautions employed at all times.

It is necessary to purge the system with an inert gas after it has been exposed to air, and before it is used for any hazardous process gas such as  $\text{PH}_3$  and  $\text{AsH}_3$ . Purging is also required to remove the process gas before opening the system to air again.

Purging is not necessary or desirable at other times. Each time a transition is made from purge gas to process gas or process gas to purge gas, there is a mixing of the two gases throughout the system; any oxygen or moisture in the purge gas may react and can be left behind as contamination. The practice of purging lines between runs gives two of these mixing events per run, causing a cumulative buildup of contamination which may eventually create problems. With a leak-tight system, there is no reason not to leave the process gas in the lines and avoid the buildup caused by the repetitive purging ritual.

It is necessary to thoroughly purge the cylinder connection line before and after opening it to the air while changing cylinders. It is not necessary to expose the entire system to purge gas during this operation. The procedures outlined in the following sections permit cylinder changing with a minimum of system exposure to purge gas.

### **3.1 Procedure to Connect a Gas Cylinder at Initial Start Up Following Facilities Installation of Purge System:**

1. Connect the 316SS tubing connection to the gas cylinder outlet

valve and tighten the CGA nut.

2. The air-operated **main shut off valve** located downstream of the flow limiting valve is normally closed (NC). Open the air-operated main shut off valve by applying nitrogen pressure (60-80 psi) to the actuator. The actuator is triggered by manually pushing the corresponding MPS-190 reset button on the annunciator panel.
3. Open the by-pass of the **flow limiting valve** as follows:  
turn counter-clockwise (CCW) one half turn from closed position.  
Use the Allen key knurled knob attached to the face of the purge panel.
4. Open the DV26 isolation valve at the inlet to the **pressure regulator**. This valve is shaped to indicate clearly the two operating positions: open when the lever is in line with the ports; closed when the lever is at right angle with ports line.
5. Turn the knob of the TDR 450 pressure regulator clockwise (CW) against its stop.

### **3.2 Procedure to Helium Leak Check MPS-190 Purge System:**

1. Open the **process valve** at the outlet to the **pressure regulator**.
2. Open **isolation valve**, **flow limit valve**, and **main shut off valve**.

3. Close the  $\text{AsH}_3/\text{PH}_3$  gas tank.
4. Initiate evacuation of the MPS-190 purge panel by applying a source of "clean" vacuum through the process valve. Helium leak check the MPS-190 system components at maximum sensitivity.

### 3.3 Procedure to Pressurize MPS-190 Purge Panel with $\text{N}_2$ :

1. Isolate MPS-190 purge system from vacuum source.
2. Supply regulated  $\text{N}_2$  (80 PSI) to purge inlet (i.e., through **purge valve**).
3. Make 20 purge cycles, each cycle consisting of the following steps:
  - a. Open **purge valve**, hold open until the pressure reaches 1 atmosphere.
  - b. Hold open for 10 additional seconds.
  - c. Close **purge valve**.
  - d. Open **vent valve**, hold open for 10 seconds.
  - e. Close **vent valve**.

f. Repeat (b-c) 20 times.

4. Close process valve at the outlet to the pressure regulator.

### **3.4 Procedure to Pressurize MPS-190 Purge Panel With Cylinder Gas Supply:**

1. Turn the knob of the pressure regulator counterclock-wise (CCW) to zero setting.
2. Open slowly the valve on the gas cylinder.
3. Close the by-pass of the flow limiting valve: turn screw clockwise (CW) to shut tight.

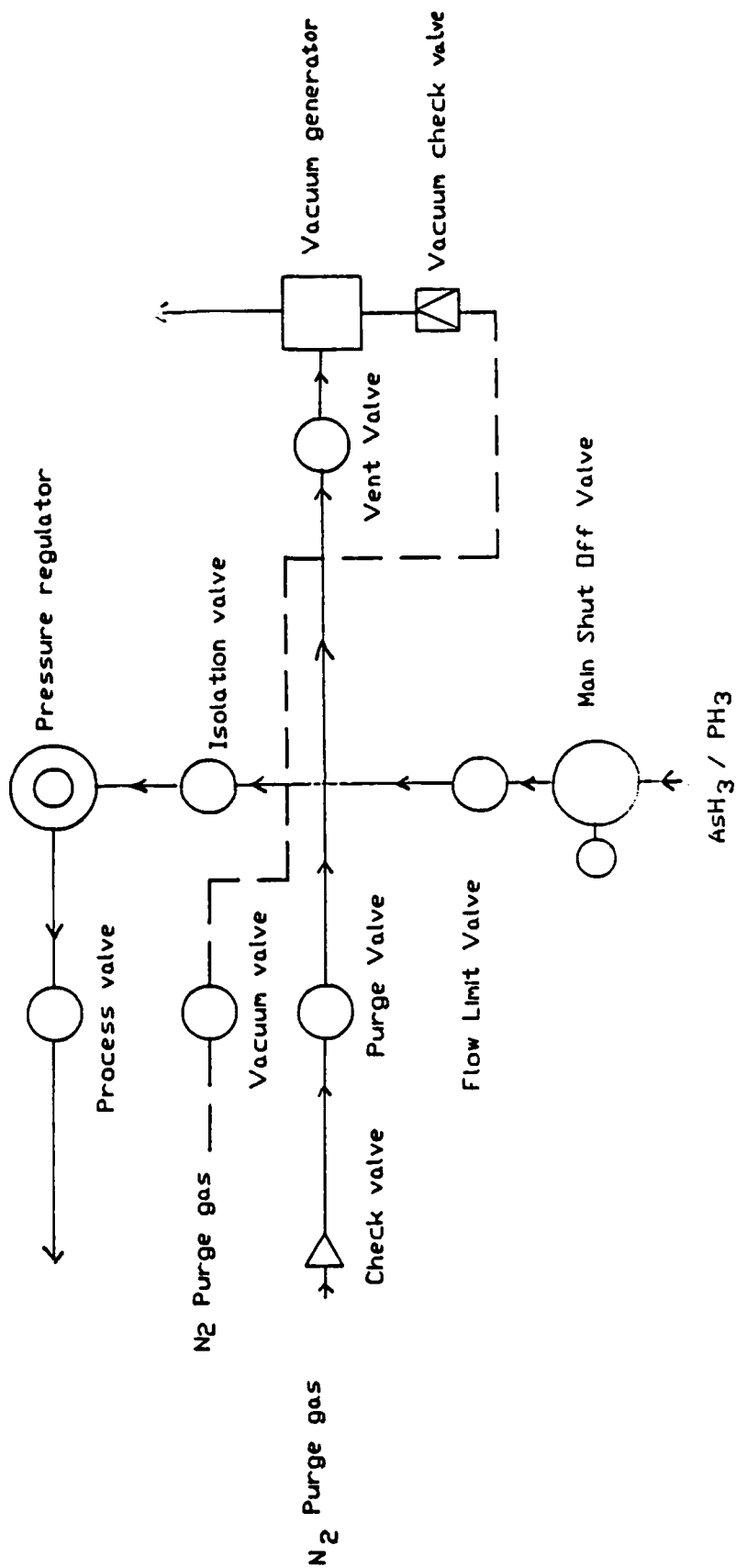
### **3.5 Procedure to Disconnect a Gas Cylinder to An Operative MPS-190 Purge Panel Installation:**

1. Close the valve on the gas cylinder.
2. Close the isolation valve.
3. Open the air-operated main shut off valve (if not already open).
4. Open the by-pass of the flow limit valve.
5. Make 20 purge cycles.

6. Disconnect the flexible connection from the valve of the gas cylinder by unscrewing the CGA fitting slowly.
7. Remove the gas cylinder.
8. Replace cylinder valve safety cap.
9. Call for cylinder pick up.

### 3.6 Procedure to Connect a New Gas Cylinder to an Operative MPS-10 Purge Panel Installation:

1. Verify that the **isolation valve** is closed.
2. Verify that the air operated **main shut off valve** is open.
3. Connect the 316SS connection to the cylinder valve and tighten the CGA fitting.
4. Apply steps outlined in sections 3.3 and 3.4.
5. Open slowly the gas cylinder valve.
6. Close the by-pass of the flow limiting valve.



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